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Research Article

SYNTHESIS AND CHARACTERIZATION OF SCHIFF BASE, 3-HYDROXY-4-(3-HYDROXY BENZYLIDENE AMINO) BENZOIC ACID AND THEIR Ni(II) AND Zn(II) METAL COMPLEXES

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ABSTRACT

A Schiff base, 3-Hydroxy-4-(3-hydroxy benzylidene amino) benzoic acid was synthesized from the corresponding starting ketone, 3-Hydroxy benzaldehyde and amine, 3-hydroxy 4-amino benzoic acid by condensation reaction. Further, the metal complexes of the Schiff base with Ni(II) and Zn(II) were synthesized. These Schiff base and its Ni(II) and Zn(II) metal complexes were characterized by spectral techniques like FT-IR, ^{1}H NMR and ^{13}C NMR spectroscopy. The synthesized compounds were evaluated for their antibacterial activity against Gram positive and negative bacteria like *Staphylococcus aureus, Bacillus subtilis*, *Escherichia coli* and *Pseudomonas aeruginosa*. The anti-fungal activities were also studied for *Candida albicans, Candida parapsilosis*. The Zn(II) metal complex exhibited the good antibacterial and antifungal activity among the all prepared compounds.

Keywords: Schiff base, Ni(II), Zn(II), 3-Hydroxy Benzaldehyde, 3-hydroxy 4-amino benzoic acid, Biological activity.

1. INTRODUCTION

From many years, the biological applications of a large number of Schiff bases and its metal complexes are investigated and studied. The imine function group containing compounds that are synthesized by condensation between an aldehyde or ketone with amine constitutes the Schiff base compounds [1]. Schiff bases are essential chemical compounds in several domains such as inorganic, analytical, and medicinal chemistry because of their flexibility, when coordinated with different transition metal ions, they may create numerous, varied, and stable complexes. Due to their diverse uses and chemical activity, such metal complexes containing Schiff bases have been widely researched in recent years. Because of their wide variety of pharmacokinetic characteristics and significance in drug development programs, Schiff base derivatives constitute a large group of molecules that have found many uses in medicinal chemistry. Schiff base derivatives and their complexes are said to have anti-inflammatory, antibacterial, antifungal, antiviral, anticancer, and antioxidant effects [2-4]. Schiff bases have also been employed in industry as very effective sensors and catalysts, as well as useful compounds for environmental preservation [5]. Several studies have shown that Schiff bases and their transition-metal complexes have biological activities that are efficient against a variety of bacterial and fungal species, as well as tumors.

One of the most fascinating aspects of coordination chemistry is the chemistry of transition metal ions when they interact with biological molecules. Coordination chemistry, or the study of Schiff base ligands interacting with metal ions, has remained one of the most active research topics in inorganic chemistry. Organic and inorganic molecules are linked via coordination compounds. Researchers have been paying close attention to Schiff bases as Nitrogen, Sulphur, Oxygen donor ligands in coordination with a variety of transition metal ions because of their notable biological applications in anticancer, antitumor, analgesic and anti-inflammatory, antibacterial and antifungal activity etc. [6].

Based on the importance of Schiff bases and their metal complexes here we report a method for the synthesis of new a Schiff base namely 3-hydroxy-4-(3-hydroxy benzylidene amino) benzoic acid and its Ni(II) and Zn(II)

metal complexes, characterized and studied the antimicrobial activity of both Schiff base and its metal complexes. Results showed that metal complexes showed more activity compared to the Schiff base.

2. MATERIAL AND METHODS

2.1. Synthesis of Schiff base ligand

The ligand was prepared by a general procedure [7]. A mixture of 3-Hydroxy Benzaldehyde (0.01 mol) and 4-amino-3-hydroxybenzoic acid (0.01 mol) was dissolved in ethanol and refluxed for 8 hrs. at a temperature of 60°C with constant stirring, in the presence of glacial acetic acid. The yellow-colored precipitate obtained was removed by filtration, washed several times with ethanol and dried. Purity of compound was checked by TLC (Fig. 1).

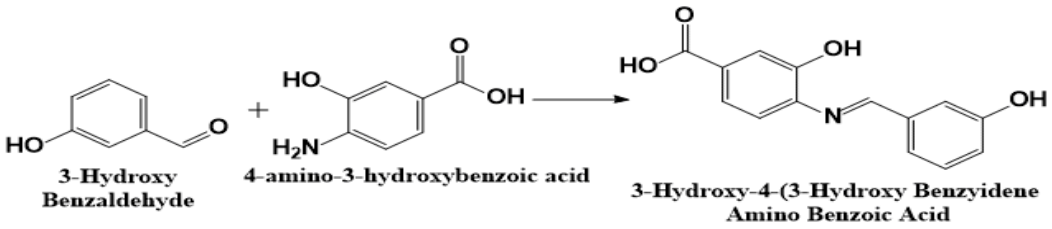

Fig. 1: Representation for the synthesis of Schiff base

2.2. Synthesis of metal complexes

Hot methanolic solution of the ligand (0.1 mol) and hot methanolic solution of corresponding metal salts (0.05 mol) (MX_2 where M= Ni(II), Zn(II); X= Chlorides) were mixed together with constant stirring and refluxed for 3 hrs. at 60°C. On cooling, coloured solid metal complexes were precipitated out. The products were filtered, washed with cold methanol and dried.

3. RESULTS AND DISCUSSION

The FT-IR spectra of prepared compounds was recorded using Bruker Alpha spectrometer. The ^{1}H and ^{13}C NMR spectra was obtained using JEOL, JNM ECS-400 Model.

3.1. Infrared spectra

The recorded IR spectra of synthesized Schiff base was as shown in the Fig. 2. The assignment of main stretching frequencies to the groups in the Schiff base was explained as follows. The vibrational frequency observed at 1551 cm^{-1} can be assigned to the υ(C=N) azomethine group which indicates the successful condensation of amine and carbonyl groups in the staring compounds which resulted in the formation of a Schiff base. The broad band centered at 3360 cm^{-1} was due to -OH groups present in the compound [8]. The aromatic C=C stretching frequency was visible at 1458 cm^{-1} with medium intensity. The aromatic C-H stretching frequency was merged with -OH frequency that gives a broad band. Form the IR data, it was concluded that the successful preparation of Schiff base. All the FT-IR spectra's resonance peaks were in line with the reported literature resonance peaks for Schiff base formation [9].

3.2. ^{1}H NMR Spectra

The proton NMR spectra of the prepared Schiff base is presented in the Fig. 3. The broad band around the 11.5 ppm can be assigned to carboxylic hydrogen atom [10]. The phenolic hydrogen was seen at 5.45 ppm. A sharp singlet observed at δ 8.59 ppm can be due to the azomethine proton and this proton confirms the successful condensation [11]. The chemical shift values in the aromatic region from 6.8 ppm to 7.8 ppm can be assigned to aromatic protons in the Schiff base which is usual for aromatic protons and comparable with reported literature values [12]. From the proton NMR results, it was concluded that the successful formation of Schiff base by condensation of amine with aldehyde.

3.3. ^{13}C NMR Spectra

The ^{13}C NMR spectra is shown in the Fig. 4. The carbon of the carboxylic acid can be seen at 168 ppm [13]. The important signal arises due to formation of azomethine group can be seen at 156 ppm and the chemical shift value is matching with the literature values [14]. The aromatic carbons of the phenyl rings can be seen in the range of 110 ppm-135 ppm [15]. These results demonstrated that the successful preparation of the Schiff base. The obtained chemical shift values are nearly same as previous reported literature and based on this chemical shift values, the Schiff base formation was successful.

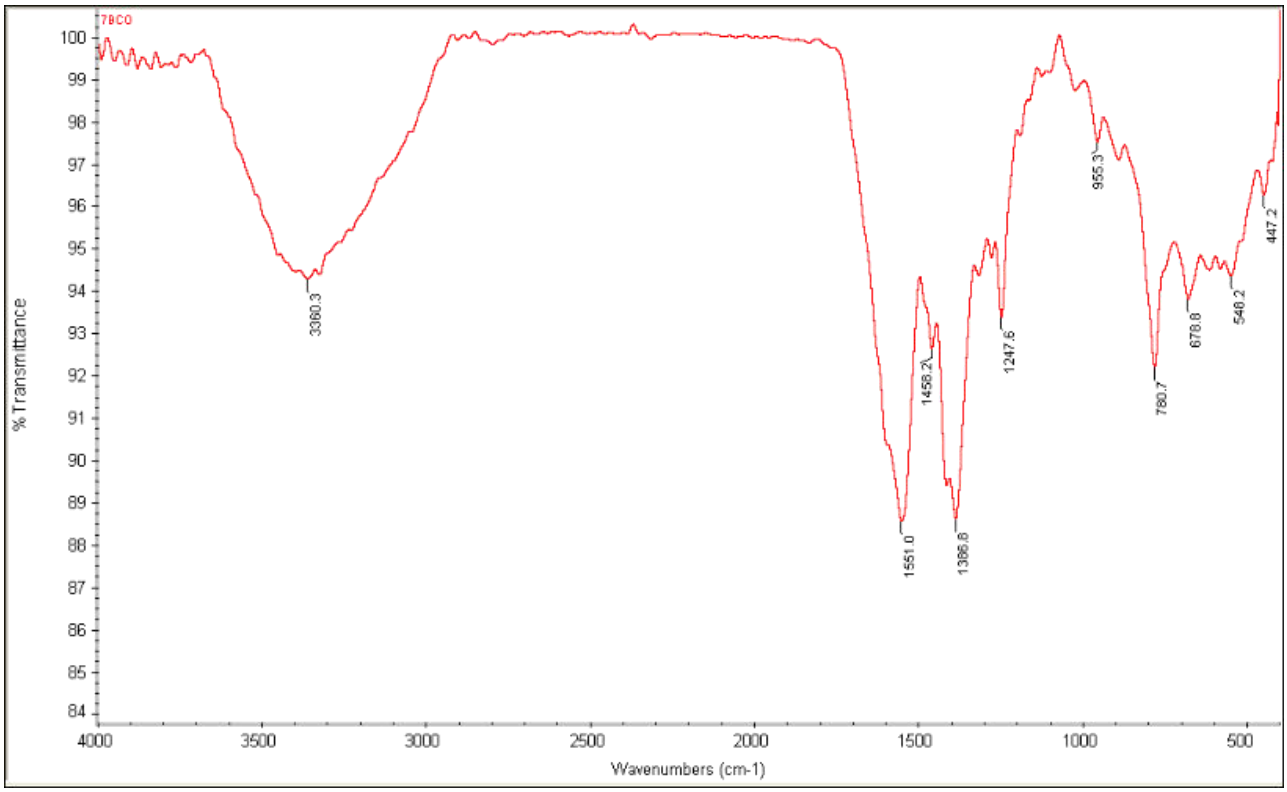

Fig. 2: IR spectra of Schiff base ligand

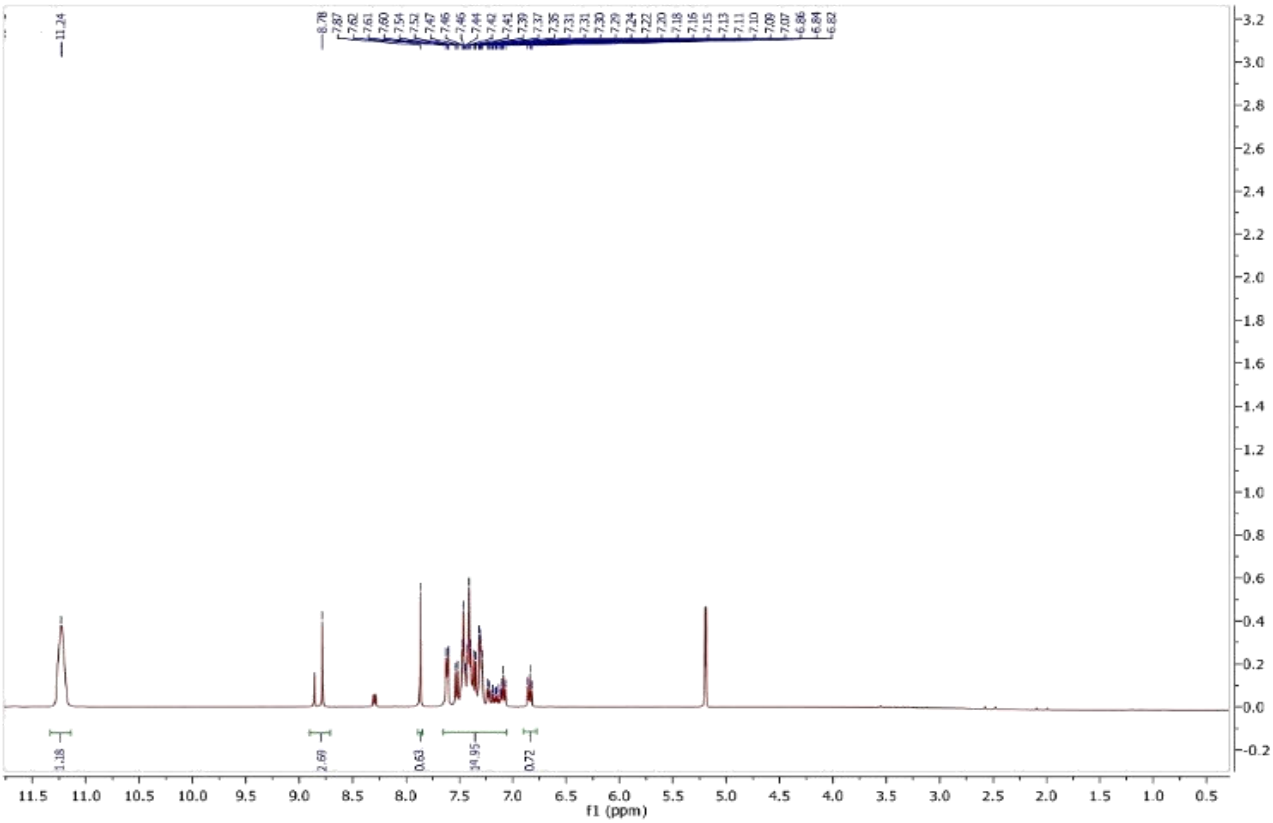

Fig. 3: ^{1}H NMR spectra of Schiff base

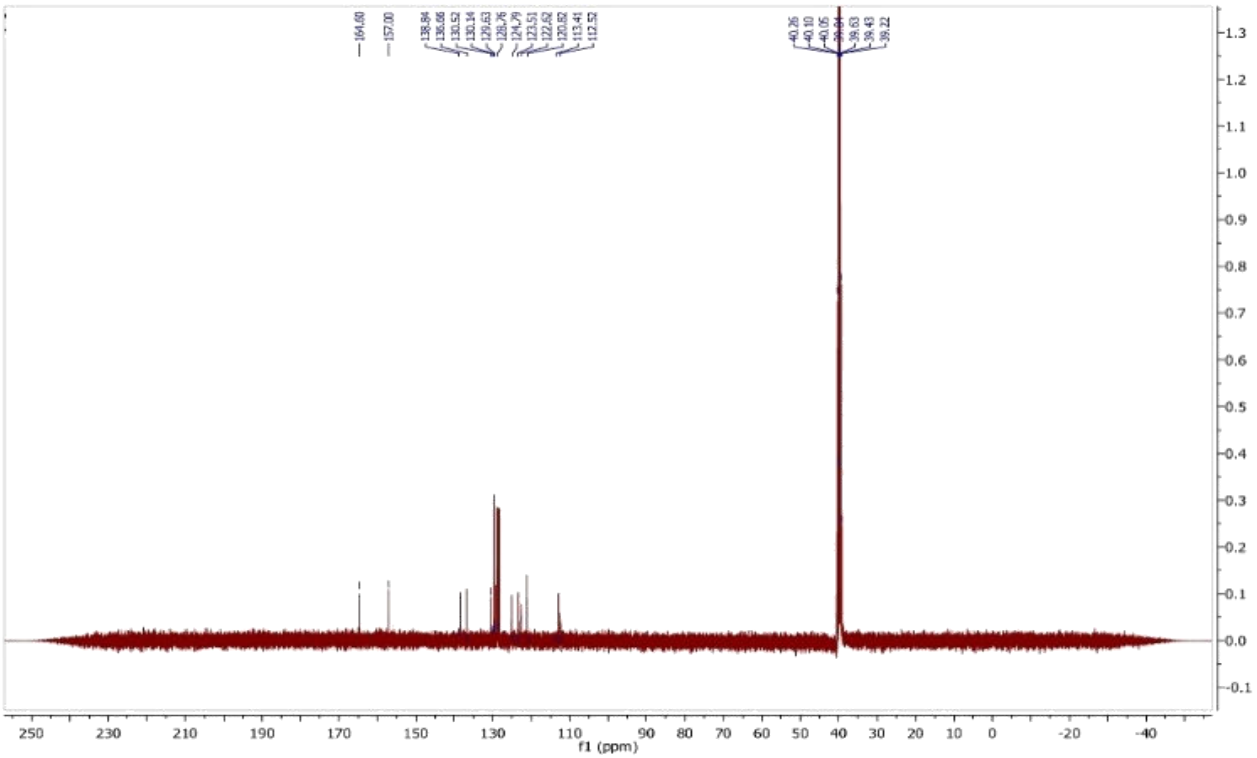

Fig. 4: ^{13}C NMR spectra of Schiff base

3.4. Antibacterial studies

The synthesized compounds are tested for their antibacterial activity against gram positive (*Staphylococcus aureus, Bacillus subtilis*) and gram negative (*Escherichia coli, Pseudomonas aeruginosa*) bacteria. Tetracycline was the Standard antibiotic. Solutions of the compounds and reference drug were dissolved in DMSO. The antibacterial activities were given as zone of inhibition in mm values. Table 1 showed that the metal complexes of Ni(II), Zn(II) are more active than the free ligand. Among the metal complexes Zinc complex of the ligand showed higher activity than the other complex. The Gram positive bacteria was most inhibited by the prepared compounds than the Gram negative bacteria because of the difference in the structure of the cell walls. The prepared Schiff base and its metal complexes have the good antibacterial values and the zone of inhibition values are correlates with the literature values [16-18].

3.5. Antifungal studies

The anti-fungal activities of the synthesized chemical compounds were tested against two yeast stains, *Candida albicans and Candida parapsilosis*. Fluconazole was used as the antifungal agent. Solutions of the test compounds and reference drug were dissolved in DMSO. Data in Table 2 shows that the inhibition values of the complexes of Ni(II), Zn(II) are higher than that of the corresponding free ligand. This indicates that the complexation process increased the fungicidal potency on two tested fungi. The results were good agreement with the literature reported values of antifungal activities of different Schiff base ligands. The higher antimicrobial activity of the metal complexes compared to Schiff base ligand can be explained in terms of chelation which makes metal complexes to act as more powerful and effective antimicrobial agents, thus inhibiting the growth of the microorganisms [19].

Table 1: Antibacterial activity studies of Schiff base

Compound	*S. aureus*	*Bacillus*	*Pseudomonas*	*E. coli*
Ligand	11 mm	14 mm	10 mm	11 mm
$Ni(L)_2$	-	-	07 mm	06 mm
$Zn(L)_2$	10 mm	18 mm	08 mm	16 mm
Tetracycline	10 mm	15 mm	10 mm	12 mm

Table 2: Antifungal activity studies of Schiff base

Compound	*Candida albicans*	*Candida parapsilosis*
Ligand	07 mm	06 mm
$Ni(L)_2$	-	05 mm
$Zn(L)_2$	09 mm	08 mm
Fluconazole	05 mm	05 mm

4. CONCLUSION

Condensation of 3-hydroxy Benzaldehyde with 4-amino-3-hydroxy benzoic acid yielded a new Schiff base, 3-Hydroxy-4-(3-hydroxy benzylidene amino) benzoic acid. Further, the chelation between Schiff base and Ni(II), Zn(II) ions yielded mono nuclear complexes. The structures of the prepared compounds were elucidated by like FT-IR spectroscopy, ^{1}H &^{13}C NMR spectroscopy. The coordinating sites of the Schiff base that are phenolic oxygens and azomethine nitrogen which chelates with the metal ions. The prepared compounds exihibited the good antimicrobial activities towards the tested bacteria. Among the all prepared compounds, the Zn(II) complex shows more antimicrobial activity than Schiff base ligand and Ni(II) metal complex.

5. ACKNOWLEDGEMENTS

Conflicts of interest

There are no conflicts of interest

Source of funding

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6. REFERENCES

1. Maurya RC, Chourasia J, Rajak D, Malik BA, Mir JM, Jain N, Batalia S. *Arab. J. Chem,* 2016; **9:**1084-1100.
2. El-Saied FA, Salem TA, Shakdofa MME, Al-Hakimi AN. *Appl. Organomet. Chem,* 2018; **32:**e4215.
3. Azam M, Al-resayes SI, Trzesowska-Kruszynska A, Kruszynski R, Shakeel F, Soliman SM, Alam M, Khan MR, Wabaidur SM, *J. Mol. Struct,* 2020; **1201:**127177.
4. Bhaskar RS, Ladole CA, Salunkhe NG, Barabde JM, Aswar AS. *Arab. J. Chem,* 2020*;* **13:**6559-6567.
5. Radha VP, Chitra S, Jonekirubavathi Shung IM, Kim SH, Prabakaran M. *J. Coord. Chem,* 2020; **73:**1009-1027.
6. Vanitha S, Kumar NS, Hari B, Reddy AV, Saritha N. *J. Med. P'ceutical Allied Sci,* 2022; **11Suppl 1:** 4470 - 4476.
7. Kailas KH, Sheetal JP, Anita PP, Apoorva HP. *World Journal of Pharmacy and Pharmaceutical Sciences,* 2016; **5 Suppl 2:**1055-1063.
8. Zoubi WA, Kandil F, Chebani MK. *Spectrochim. Acta A Mol. Biomol. Spectrosc,* 2011; **79 Suppl 5:**1909-1914.
9. Tyagi P, Chandra S, Saraswat BS. *Spectrochim. Acta A Mol. Biomol. Spectrosc,* 2015; **134:**200-209.
10. Tandon JP, Crowe AJ, Road F. *Polyhedron,* 1986; **5 Suppl 3:**739-742.
11. Hammam AM, Khafagi ZA, Ibrahim SA. *J. Mater. Environ. Sci,* 2015; **6 Suppl 6:**1596-1605.
12. Santos JE, Dockal ER, Cavalheiro ÉTG.*Carbohydr. Polym,* 2005; **60:**277-282.
13. Manjula B, Arul S. *Asian Journal of Biochemical and Pharmaceutical Research,* 2003; **1:**168-178.
14. Canpolat E, Yazici A, Kaya M. *J. Coord. Chem,* 2007; **60 Suppl 4:**473-480.
15. Tyagi P, Chandra S, Saraswat BS. *Spectrochim. Acta A Mol. Biomol. Spectrosc,* 2015; **134:**200-209.
16. Alahdal AM, Asfour HZ, Ahmed SA, Noor AO, Al-Abd AM, Elfaky MA, Elhady SS.*Molecules,* 2018; **23 Suppl 4:**978-986.
17. Mohapatra RK, Mishra UK, Mishra SK, Mahapatra A, Dash DC. *Journal of the Korean Chem. Soc,* 2011; **55 Suppl 6:**926-931.
18. El-Sherif AA, Eldebss TMA. *Spectrochim. Acta - A: Mol. Biomol. Spectrosc,* 2011; **79 Suppl 5:**1803-1814.
19. Abu-Dief AM, Abdel-Rahman LH, Abdel-Mawgoud AAH. *Appl. Organomet. Chem,* 2020; **34 Suppl 2:**e5373.